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Morphological evolution of the fivefold surface of *i*-AlPdMn quasicrystals

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Morphology of the fivefold symmetric quasicrystal surface of AlPdMn was investigated by x-ray reflectivity and by x-ray diffraction. X-ray experiments revealed two different morphologies depending on the surface preparation. Sputtering and annealing up to 900 K, under UHV conditions, produced a rough and facetted quasicrystal surface. These features were confirmed by atomic force microscopy and scanning tunnel microscopy measurements. We also observed that an annealing above 900 K induces a rapid and irreversible transition toward a flat surface.

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I. INTRODUCTION

Quasicrystals present very peculiar and attractive tribological properties, such as low adhesion, weak friction, and a *nonstick* character.[1–3] These appear to be intrinsic surface properties of quasicrystals, even if the origins are not easily identified. Experimental observations revealed a surprisingly low surface energy for quasicrystals, when a thin layer of aluminum oxide is present at the surface.[1] These specific adhesive properties are gradually lost if the thickness of the aluminum oxide layer is increased. These observations could be correlated to the intrinsic electronic properties of quasicrystals, which show a pseudogap[4] at the Fermi level and the noticeable absence of a Drude behavior.[5] Together with adhesive properties, quasicrystals showed a very low friction coefficient where a wear regime is predominant.[2] Tribological measurements were carried out either on quasicrystal films and polished surfaces exposed to the air[3] or between two clean quasicrystal surfaces, prepared under UHV conditions.[2] Quasicrystal/quasicrystal contact for AlPdMn exhibited neither self-adhesion nor a *stick and slip* behavior. In the same experimental conditions, the friction coefficient is not defined for the Cu/Cu contact, owing to strong stick and slip behavior.

The Frenkel-Kontorova model[6] highlights a fundamental link between incommensurate interfaces and low friction, in the limit of weak interaction.[7,8] This model has recently been applied by Lançon *et al.*[9] in order to explain the vanishing of static friction between two quasicristal surfaces. However, it is not entirely clear whether aperiodic structure and surface morphology are responsible for the tribological properties mentioned above. The lack of the Bravais lattice implies that the terrace-ledge-kink model[10] cannot be easily transferred to the quasicrystal surface. A new kind of morphology appearing in quasicrystals could decrease the contact area between two quasicrystalline surfaces and reduce the friction coefficient.

In order to understand the role of the morphology in tribological properties, it is first necessary to establish a relationship between the structure of the quasicrystal and its morphology, in the absence of the Bravais lattice. As these properties involve surface phenomena, they motivated fundamental studies either of structure,[11–16] morphology,[17–23] or chemical reactivity[24–26] of quasicrystalline surfaces. Nowadays, the discovery of thermodynamically stable quasicrystals and the availability of several centimeter quasicrystal single grains,[27–32] combined with the ability to prepare clean, flat, and well organized surfaces, allows their properties to be completely characterized.

X-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES) ensure the efficiency of ion bombardment at room temperature and in UHV conditions, in obtaining very clean surfaces (neither oxygen nor carbon spectroscopic signatures). Nevertheless, the chemical selectivity of the bombardment induces a sever shift in the chemical composition, compared to the bulk.[14,33–37] To recover the nominal composition of the icosahedral phase, a brief annealing above 700 K is required.[15,38] Low-energy electron-diffraction (LEED) studies showed that, after several cycles of sputtering and annealing at 700 K, an aperiodic structural order appears at the surface of this alloy. The high quality LEED patterns and the position of diffraction peaks, obtained on the fivefold symmetry surface, indicate that the surface is ordered and its structure is very similar to the bulk. Eventually, the structural quality of this surface increases with the annealing temperature. Further experiments of x-ray surface diffraction[39] indicate that in order to obtain a very flat and well organized surface, an annealing at $T > 900$ K is essential. This surface shows a bulklike termination and a long-range order. This result confirmed the scanning tunnelling microscope (STM) observations by T. Schaub *et al.*[12,16,40] STM measurements showed that, after sputtering and annealing above 900 K, the fivefold symmetry surface can be described by a set of atomically flat terraces with steps of 4.1 and 6.6 Å: this description is consistent with a sequence of dense planes, distributed following a Fibonacci sequence.

There is a strong underlying physical question in this study of the fivefold surface of AlPdMn quasicrystals: what is the intrinsic long range morphology of a fivefold surface in equilibrium with the bulk? Results on the morphology and on the properties of quasicrystal surfaces are still a matter of debate although it is nowadays a rapidly evolving situation. We face the following experimental situation. On one hand, cleaved surfaces prior to any annealing treatment are well described as rough surfaces down to the nanometer scale although they remain mirror like at large scale.[22,23] This surface state can be radically changed by annealing close to 900 K with the appearance of a facetted micrometer size holes at the surface. On the other hand, sputter and annealed surfaces through local observation by STM have been shown to undergo a similar change.[15,20] When annealed at temperatures lower than 900 K, rough surfaces are seemingly observed on a nanometer scale. Organization of the surface in terrace and step structures only occurs after annealing at temperatures higher than 900 K. A major question in this field is to ascertain that this surface transformation is not only a transient state associated with a poorly organized surface (the surface diffusion on typical experimental time may not be efficient enough). Studies in our group have now tackled this problem in two directions: (i) use of x-ray reflectivity and of x-ray diffraction which both average over large areas, to demonstrate that this surface transformation takes place as a massive process; (ii) analysis by x-ray diffraction of the bulk structure in the surface vicinity (over micrometer thickness) to identify bulk changes associated with this surface change. The present paper reports on the first point. X-ray diffraction and x-ray reflectivity simultaneously shows that annealing at 900 K definitely triggers an irreversible transformation between a rough surface to a flat surface at short scale. The second aspect is now the subject of an experimental program under progress. The experiments were therefore completed by atomic force microscopy (AFM) observations, which provide local information about the morphology, from the nanometer scale up to 100 μm.

II. EXPERIMENTAL DETAILS

The quasicrystal sample used in diffraction experiments was a single grain, grown using the Bridgmann method. Its nominal composition (the initial liquid composition used in growth) was $Al_{72}Pd_{19.5}Mn_{8.5}$.[37] AFM and STM studies were performed on two different samples: the previous one and a Czochralski grown single grain, whose nominal composition (liquid composition) was $Al_{68}Pd_{21}Mn_{11}$.[29,41,42]

Samples were cut with a fivefold axis normal to the surface with a precision of 0.1°. Surfaces were mechanically polished to a mirrorlike surface.

The samples were cleaned by cycles of ion sputtering and annealing in UHV, as described elsewhere.[35,36] The efficiency of this treatment was verified for each sample, either by LEED or by x-ray photoelectron spectroscopy (XPS).[26,37,40,43] The upper temperature limit was chosen to avoid phase transformations.[39]

X-ray studies were carried out at the ID32 beam line at ESRF. This beam line, conceived for surface studies, is equipped with an UHV chamber coupled to a four-circle diffractometer. That allows sample preparation and characterization *in situ*. As in this chamber, for geometrical reasons, a LEED facility was not available, surface reorganization has been followed by medium-energy electron diffraction (MEED). Neither the energy (6 keV) nor the incidence angle ($\sim$20°) of the electron beam makes the MEED a technique very sensitive to the surface order, because the penetration depth of electrons exceeds a few nanometers. However, we were able to verify that the MEED pattern appeared only after several sputtering-annealing cycles and it disappeared as the surface was bombarded: this behavior was consistent with LEED and XPS measurements.

The x-ray wavelength was selected by means of a double-crystal monochromator, using the reflection (111) of silicon. Its relative $\Delta\lambda/\lambda$ resolution is about 10^{-4}. For this experiment the wavelength was tuned to 1.2425 Å, which corresponds to 9.978 keV of energy. At this energy, the penetration depth of x rays is less than 100 Å at grazing incidence angle.

AFM measurements have been performed in air at room temperature: the samples were prepared as described above, under UHV conditions. Afterward the samples were exposed to the air. In contact with the air, a very thin and stable layer of aluminum oxide (2 nm thick) immediately grows on the quasicrystal.[44] We believe that ambient air contact does not affect the morphology on a large scale, compared to the oxide layer thickness. Experimental results presented in the following figures were obtained in *contact mode*. Nanometer scale roughness was characterized by STM under UHV conditions, immediately after surface preparation.

III. EXPERIMENTAL RESULTS

A. X ray measurements

Figure 1 presents the x-ray reflectivity of a quasicrystal surface. Measurements were carried out at room temperature after several cycles of sputtering and annealing. Reflectivity has been measured using a $\theta-2\theta$ scan at grazing incidence, for two different annealing temperatures. At the beginning the sample was gently annealed, in order to obtain a good MEED pattern. The temperature was limited to about 750 K and the preparation cycles lead to a total annealing time of more than one hour. No changes were observed in the reflectivity shape as a function of the annealing time. Figure 1(a) shows the x-ray reflectivity measured on this surface. The intensity ratio between the reflected and the incident beam is plotted as a function of the incident beam angle. The electronic density of the quasicrystal surface determines the total reflection angle. At this energy, the calculated critical angle is 0.24° for the $Al_{70}Pd_{20}Mn_{10}$ quasicrystal, which corre-

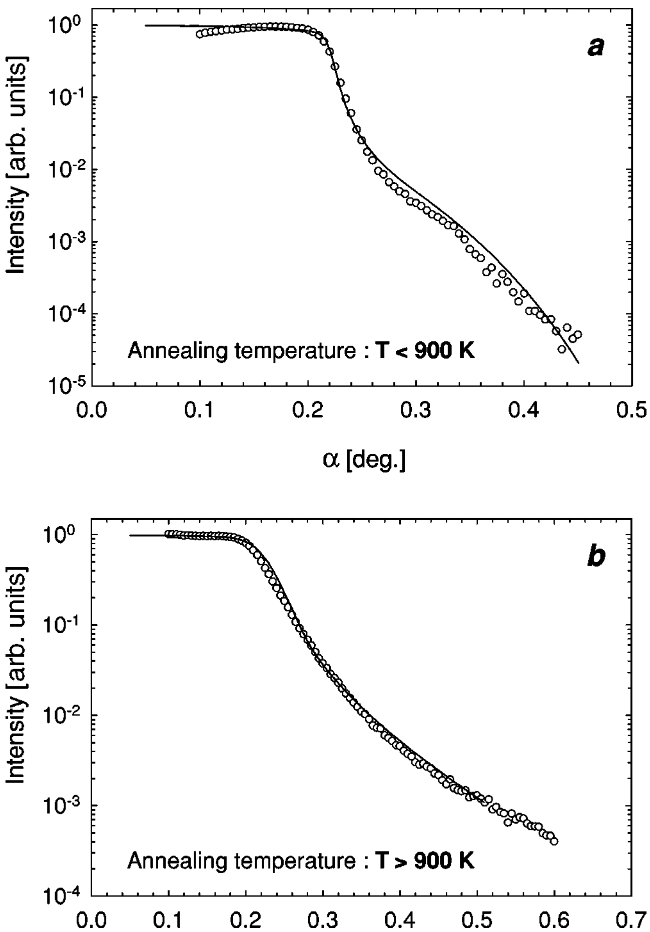

FIG. 1. X-ray reflectivity of the fivefold surface of the AlPdMn quasicrystal. α is the incident angle of the x-ray beam. Given are the measured data and continuous lines are calculated reflectivities. (a) Reflectivity after annealing at $T<900$ K, (b) reflectivity after annealing at $T>900$ K.

sponds to the measured reflectivity (open circles), if one takes in account the absorption effects. Compared to the theoretical reflectivity, calculated for a flat and homogeneous surface, the experimental curve presents an anomalous bump at about 0.3°. Such a bump can be classically assigned to a change in the surface density. A homogeneous layer, with a thickness of about 5 nm and density decreased by 10% can explain this result. The correlation is even better if roughness is included at surface and interfaces. The continuous line represents the simulated reflectivity curve with the thickness and density mentioned above[45] and a surface/interface roughness RMS of 1 nm (within the framework of the Croce-Névot model[46]).

However, such a change in density should lead at metallic atomic densities to an important increase of the aluminum composition. After this surface preparation, the chemical composition measured by Auger and XPS is close to the bulk and does not suffer important variations upon annealing at even higher temperatures. These two experimental conclusions are therefore not consistent. The bump in surface reflectivity can be attributed more satisfactorily to a short scale surface roughness.

Figure 1(b) presents the reflectivity curve on the same sample after a short annealing (few minutes) at temperature close to 900 K. The striking and reproducible difference with the Fig. 1(a) is that the previous bump has been completely and irreversibly erased. Furthermore, the continuous line is a simple fit that is based on the flat surface of a bulk terminated AlPdMn quasicrystal surface. The agreement between experimental data and the theoretical reflectivity curve is better if a surface roughness of about 6 Å (RMS) is introduced.

Further cycles of sputtering and annealing at lower temperature (750 K) did not reproduce the previous surface. This result indicates that the annealing at 900 K produces a severe and irreversible evolution of the surface.

In x-ray surface diffraction the regions of lower intensity between two bulk Bragg peaks, along the direction perpendicular to the surface, are the most sensitive to the surface structure and morphology. In fact, a surface can be considered as the truncation of an infinite crystal. This truncation introduces a diffuse intensity in the diffraction signal out of Bragg position.[47]

While in periodic infinite crystals no signal is expected out of discrete Bragg position, in quasicrystals peaks are dense in the reciprocal space. This introduces an additional difficulty in distinguishing surface and bulk signal.

With the intention of differentiating the surface signal from the contribution of weak bulk Bragg peaks, the truncation rods of the annealed surface are systematically compared to the rods of the sputtered surface. Ion sputtering is supposed to introduce structural disorder and roughness at the surface. Consequently, we expect a drastic decrease of diffuse signal out of Bragg position after the bombardment.

Figure 2 shows the measured specular $00L$ crystal truncation rod ($\theta-2\theta$ scan) perpendicular to the fivefold surface. Figure 2(a) corresponds to a surface sputtered and annealed at $T<900$ K, while Fig. 2(b) represents the truncation rod for the same surface annealed at more than 900 K. Each point of this curve is the result of the integration of transverse scan (α scan).

In Fig. 2(a) the $00L$ rod scan after sputtering is represented by filled circles and by open circles after annealing at $T<900$ K. No differences are observed between the curve acquired after the bombardment and after the annealing. Several cycles of sputtering and annealing lead to a total annealing time of more than one hour, without any change in the $00L$ rod. In the same preparation conditions, XPS and LEED measurements probed that both the structure and the chemical composition are consistent with a quasicrystal surface. We could conclude that this surface is not flat enough over a large distance to diffuse x rays coherently.

Figure 2(b) represents the $00L$ truncation rod after a short annealing above 900 K (open circles), and the same rod measured after an additional ion bombardment (filled circles). The comparison highlights a clear surface signal, characteristic of a flat surface.

An in-plane scan, across a *nonsymmetric* truncation rod out of Bragg position, confirms this result. Figure 3 shows the dramatic decrease of the intensity between Bragg peaks, as the quasicrystal surface is bombarded: ion bombardment at 3 keV for several minutes increases the surface roughness.

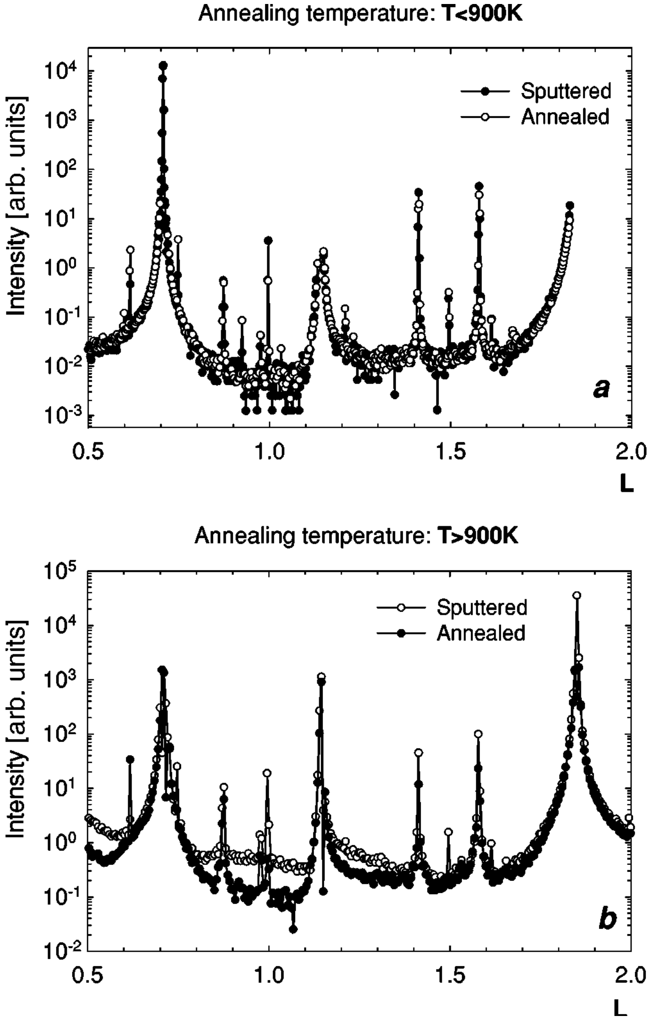

FIG. 2. Specular crystal truncation rod along a fivefold axis. (a) after ion sputtering (filled circles) and after annealing (open circles) at $T<900$ K, (b) after sputtering (filled circles) and after annealing at $T>900$ K (open circles).

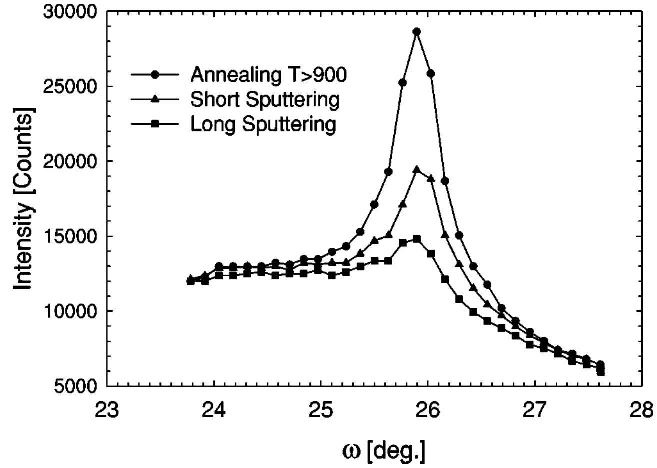

FIG. 3. Transverse scans (ω scan) at the minimum between two Bragg peaks. Filled square symbols correspond to the sample annealed at 900 K. The peak disappears progressively with the sputtering time (lower curves).

The peak width probes the structural quality of this surface. After annealing at high temperature, the width of 0.02 Å^{-1} corresponds to an in-plane structural coherence of about 300 Å.[47] This value agrees with other results, obtained by x-ray diffraction (Alvarez *et al.*[39]) and by high-resolution LEED (P. Thiel *et al.*[48]), both with analogous surface preparations. In x-ray diffraction the regions out of Bragg position are the most sensitive to the surface morphology. Figure 4 shows transverse scans across the 00L rod, for different values of L near to the Bragg peak located in $L=0.71$ [first peak in Figs. 2(a) and (b)]. The same experiments were carried out both after annealing at 750 K [Fig. 4(a)] and on the surface annealed at 900 K [Fig. 4(b)].

We observe in Fig. 4(a) that the surface annealed at low temperature reveals a double peak, out of Bragg position on both sides of the 00L rod, whereas no signal is observed along the L direction. Both peaks have approximately the same intensity and their distance Δh increases proportionally with the distance from the Bragg peak. The inset in Fig. 4(a) shows that peaks are aligned along two lines, intersecting each other at the Bragg position. This result is consistent with a facetted surface, where a truncation rod originates in each facet. The angle between facets is related to the intersection angle of the rods, which has an experimental value of 4.3°.

A short annealing at 900 K completely erased the double peak, as showed in Fig. 4(b). Transverse scans across the specular rod indicate that only one peak exists along the L direction, instead of the double peaks observed before the high-temperature annealing. This measurement is consistent with the evolution of reflectivity and 00L rod, and it confirms that two characteristic surface morphologies exist.

The surface produced at high temperature is flat and characterized by steps and terraces. It is certainly the most investigated surface in the field.[16,20,39,40,43] The surface obtained after annealing below 900 K is rough for x-ray diffraction but is associated with the LEED pattern.[20,21,40] It may be argued that the classical problem of insufficient surface preparation is the reason for this observed behavior and that a homogeneous surface state is reached only at high temperatures. It is difficult to completely disprove this point. In our experiments, long annealing at moderate temperature does not change the surface morphology. To the contrary, a temperature annealing at 900 K leads to rapid irreversible flattening of the surface.

B. AFM/STM measurements

On the basis of the reflectivity behavior (Fig. 1) and results from x-ray diffraction (Figs. 2, 3 and 4), we have investigated the morphology of fivefold surface of samples annealed at low temperature (between 700 and 900 K) using AFM. The initial surface was optically polished, with a peak to peak roughness of about 100 nm. Figures 5 and 6 show the surface morphology perpendicular to a fivefold axis, after cycles of bombardment and annealing. As shown in Fig. 5, this surface preparation produces a large-scale structure organized with high steps (macrosteps with height close to 100

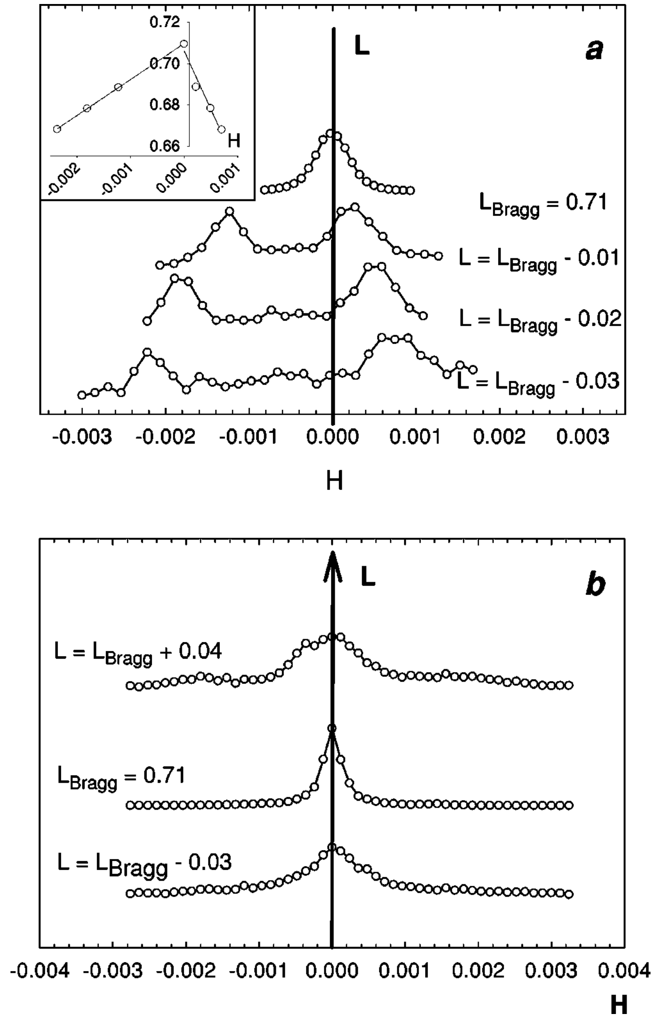

FIG. 4. Transverse scans (α scan) across the specular truncation rod. (a) The surface annealed at $T<900$ K reveals a double peak out of Bragg position. Distance between the peaks increases with the distance from the Bragg position. (b) After annealing above 900 K, a single peak appears instead of the double peak.

nm) and terraces. Terraces (areas with constant gray level in Fig. 5) are large and parallel to each other. At this micrometer scale, the macrosteps are sharp, perpendicular to the surface, and straight over more than 1 mm and with limited roughness along the step. Comparison of the two large holes in Fig. 5 (dark area close to the image center) reveals the distinctive features of this surface. The macrosteps on the surface only present well defined orientations. The hole presents five straight and long steps separated by five shorter edges which are not well defined steps. If the step lengths were identical, the five steps would make up a regular pentagon, as it is depicted in the inset of Fig. 5. These orientations appear to be constrained by the bulk order of this single grain (fivefold symmetry axis perpendicular to the surface).

Therefore annealing at temperatures lower than 900 K leaves a simple surface structure, characterized by steps and terraces at micrometer scale. At a shorter scale, we still observe terraces and steps, but both are now characterized by a high roughness. The surface in Fig. 6 presents a step/terrace

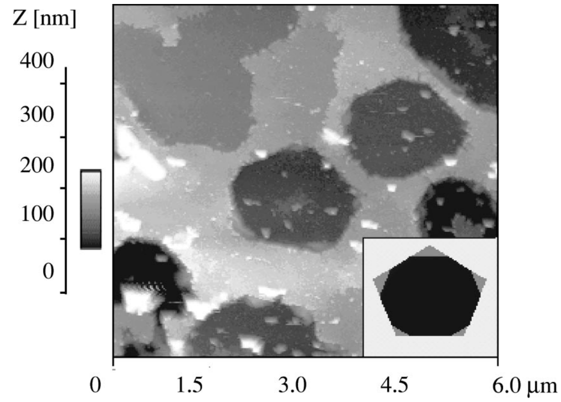

FIG. 5. Air-AFM image on the fivefold surface of AlPdMn quasicrystal after sputtering and annealing at 850 K. Large scale structures (6×6 mm). Holes (dark area) with depth of 100 nm are observed. The hole at the center has a pentagonal symmetry as comparison with schema in inset shows.

structure, with a step height close to 10 nm. However, the main feature is that the terraces appear as packing of small spheres of about 10 nm radius, which is very close to the step height. It implies that the steps are not very well defined, as a one-dimensional line. Figure 6 suggests that it could be more pertinent to define a step height as the mean height difference between two terraces. Analysis of the terrace structure at different place reveals that this short scale roughness is a distinctive feature of this surface morphology. Therefore the cluster-based structure appears as an intrinsic feature of quasicrystal surface, prepared by sputtering and annealing up to 900 K.

These observations were completed by scanning tunnel microscopy (STM) experiments, which allow the morphology to be investigated down to nanometer scale. Surface oxide formation could prove critical, if analyzed details are smaller than oxide thickness (2 nm). In order to avoid oxidation and surface contamination, experiments were carried out *in situ*, immediately after surface preparation. Results are presented in Fig. 7. The essential aspect in this picture is the

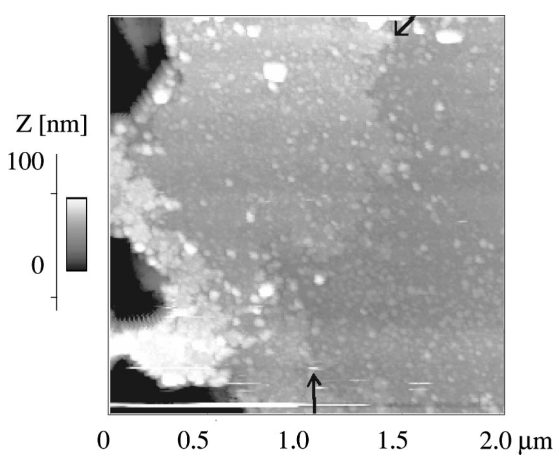

FIG. 6. Detail of Fig. 5. Black arrows identify a rough step whose height is 10 nm. The cluster structure of the terrace is observed.

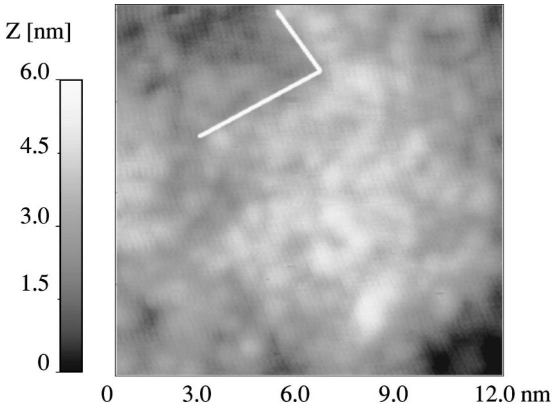

FIG. 7. STM image under UHV of the fivefold surface of AlPdMn quasicrystal, after ion sputtering and annealing at a temperature of 750–800 K. A cluster structure on a nanometer scale can be inferred from this observation.

contrast at the nanometer scale. This surface seems to be the result of nanoelement packing. This packing contributes to create a *hill and valley* structure, similar to the structure observed on cleaved surfaces.[22,23] Neither sharp steps nor flat terraces were observed up to the micron scale (upper limit of the STM scan width). On the other hand, we can observe some very rough steps, as highlighted by white lines in the upper side of the picture.

The nanometer clusters present a characteristic size close to a nanometer. They also present, at the STM resolution, a spherelike shape.

IV. CONCLUSION

Surface morphology studies on AlPdMn quasicrystals reveal a complex framework. Two completely different morphologies are observed depending on the surface preparation. X-ray reflectivity and diffraction experiments show that cycles of sputtering and annealing below 900 K produce a rough and facetted surface, while high-temperature annealing (more than 900 K) induces a rapid and irreversible transition toward a flat and well organized surface, characterized by a steps and terraces morphology.

The description deduced from both x-ray and AFM/STM experiments is that the surface obtained at low temperature is macroscopically flat on a large scale (about 1 mm) and rough on a small scale (below about 100 nm). At large scale, the morphology is essentially dominated by a step and terrace structure. The short scale roughness deduced from AFM and STM images is consistent with the x-ray observations presented in this paper. At this scale, extremely rough steps can still be identified as jumps between different layers of packed clusters perpendicular to a fivefold axis. These results show that, when annealed below 900 K, the surface presents a cluster-based organization, with definite cluster sizes and shape. Defined terraces and rough steps at large scale also coexist with clusters on this surface.

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